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SPAR V TECHNICAL REPORT FOR EXPERIMENT 74-37 CONTAINED POLYCRYSTALLINE SOLIDIFICATION IN LOW-G

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RESEARCH DEPARTMENT

SPAR V TECHNICAL REPORT FOR EXPERIMENT 74-37 CONTAINED POLYCRYSTALLINE SOLIDIFICATION IN LOW-G

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ABSTRACT

The effect of gravity on the columnar-to-equiaxed microstructural transition was studied in small samples of NH_4Cl-H_2O . The behavior of the samples during laboratory (one-gravity) simulations was contrasted with their behavior during the SPAR V (low gravity) sounding rocket flight. In one gravity, the columnar zone accounted for 25 to 100% of the structure, depending on the superheat and orientation of the chill. Grain multiplication occurred by showering and by convection induced dendrite arm remelting. Convection was caused by both thermal gradients and solutal gradients. In low gravity, completely columnar structures were obtained; all grain multiplication mechanisms were entirely supressed. The absence of gravity also modified the thermal conditions and caused the liquid to cool more slowly. This resulted in a steeper temperature gradient in the liquid ahead of the solidification interface. The growth rate of the solid was not affected. A spring-loaded expansion piston jammed, causing "big bang" nucleation in two chambers and distributing nuclei throughout the liquid. Despite this occurrence, an equiaxed zone did not form, indicating that the most significant effect of low gravity on this experiment was the modification of the thermal conditions. This resulted in a situation in which the equiaxed grains could not grow and rendered the columnar-to-equiaxed transition impossible.

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INTRODUCTION

A zone of equiaxed (roughly spherical) grain structure will form in a casting only when nuclei for equiaxed grains are present in the melt and growth of the equiaxed grains is more rapid than growth of the columnar grains (Ref. 1). Both conditions are necessary and both steps can be influenced by gravitation.

Nuclei can appear in the melt in various ways. Inoculants may be present, making nucleation easy (Ref. 2). At low melt superheat, a large number of nuclei may form during pouring and be distributed through the liquid according to local fluid flow patterns ("big bang" nucleation (Ref. 3)). Convection, caused by thermal gradients, can induce remelting and break-off of dendrite arms from the growing dendritic interface. These arms are then transported to the center of the casting and act as nuclei for the formation of the equiaxed grains (Ref. 3). Convection may also be caused by the presence of composition gradients which arise from rejection of solute, even in a nonconvective thermal field. This process has been shown to be responsible for the formation of freckles, regions of fine equiaxed grains in a directionally solidified columnar grain structure (Refs. 4, 5). Also, Southin has demonstrated (Ref. 6) that fragments of dendrites can grow at the open surface of the solidifying ingot, "shower" down through the melt, settle on the advancing columnar interface, and form equiaxed grains. Finally, constitutional supercooling of the melt ahead of the interface can, in principle, cause nucleation (Ref. 7), although this is rarely observed.

All of the above mechanisms are influenced by gravitational acceleration. Reduced gravity will slow down or surpress those mechanisms that depend on density difference, namely thermal convections, solutal convection, and showering. In the case of heterogeneous nucleation reduced gravity could enhance nucleation by preserving a more uniform distribution of inoculants, because of reduction of the Stokes' settling or floating force. As for constitutional supercooling, reduced gravity may change the solutal and thermal gradients near the interface by altering thermal and mass transport in the melt. The precise effect of a reduction in gravity on each one of these gradients is not obvious; both might be expected to be steeper, with the thermal gradient being modified more than the solutal gradient

(Ref. 8). However, valid guidelines for a priori predictions of the effects of reduced gravity on these gradients at the solidification interface have not yet been developed.

Likewise, it is difficult to predict how reduced gravitational acceleration will affect growth of equiaxed relative to columnar grains. In the only model which directly considers growth, Burden and Hunt assume that the rate of growth of the columnar interface decreases continuously thereby causing the temperature at the growth interface to go through a maximum (Refs. 9,10). As the growth rate of the columnar grain continues to fall the interface temperature decreases, thereby increasing the rate of heat transport out of the melt and increasing the growth rate of equiaxed grains, causing the columnar-to-equiaxed transition (CET). They do not consider convective effects; and an increased temperature gradient can lead to an increased or decreased growth rate depending upon which side of the maximum in the growth rate versus interface temperature curve one is located.

In a clear cut low gravity experiment using NH₄Cl solutions, Johnston and Griner (Ref. 11) showed that dendrite arm remelting caused by thermal inversion driven convection did not occur, and a completely columnar structure was obtained. Other low gravity experiments have resulted in the formation of equiaxed structures instead of the expected columnar structures (Refs. 12, 13), but these observations remained largely unexplained. The significant effects of grain structure on some mechanical properties have prompted the current investigation. The main objective of this study was to directly observe the effect of a reduction in gravity on the CET (Ref. 14). It was thought that the experiment should be designed so that the occurrence of the CET varied in a systematic fashion in the ground base simulations; it was thus expected that the primary effect of reduced gravity on CET could be established.

EXPERIMENTAL PROCEDURE

This experiment was performed in a specially built apparatus which was designed for the payload of a Black Brandt sounding rocket. During the rocket flight, a freefall interval of approximately 250s occurs in which all accelerations are less than 10^{-4} g_o (g_o = 980 cm/s²). This "low-gravity" interval begins at approximately 80s after lift-off. The apparatus was programmed to perform a solidification experiment during that interval. The progress of the low-gravity experiment was recorded by a 35 mm camera which took 230 photographs at a rate of one frame per second. The apparatus has been described in detail elsewhere (Ref. 15), thus only a brief description will be given here. The heart of the apparatus is the solidification chamber, shown schematically in Fig. 1. It consists of a semicylindrical chill block, made of copper, brazed to a top plate of stainless steel. The front and rear faces are of plexiglass and are sealed with "O" rings. This geometry was chosen to provide radial heat extraction from the samples to simulate weld bead solidification and to allow direct observation of the experiment. Four such chambers are arranged symmetrically as shown in Fig. 2. All of the chill blocks were machined from a single piece of copper. The copper web which joins the four cells serves to transmit heat to and from the cells during prelaunch soaking and during the quench. Before launch the four-sample assembly is heated to and controlled at a uniform temperature of 80° C. At launch, power to the heaters is cut off and at 100s after lift off a timer opens a solenoid valve which allows liquid freon-12 to spray on the copper web, cooling the samples. Solidification takes place during the following 200s and is recorded by the camera. Simultaneously, readings are taken from thermistors which measure the temperature of the sample material in chamber I. The thermistors are located in the front and back plexiglass faces at equal intervals of 1/4 of the radius starting at the apex of the chamber. Two additional thermistors record the temperature of the chill block. Other details of the sample chambers are illustrated by the figures in the Results and Discussion section.

Two $\rm H_2O$; $\rm NH_4Cl$ hypereutectic alloys were used; 325 grams of $\rm NH_4Cl$ per litre of solution (solution A) and 362.5 gm/litre, solution B. The $\rm NH_4Cl$ was reagent grade and the solutions were filtered after preparation. The liquidus temperatures

were 35 and 55° C, respectively. The solidus is given as -15° C by Copley, et al. (Ref. 5). The soak temperature was 80° C, thus the superheats were 45 and 25° C, respectively.

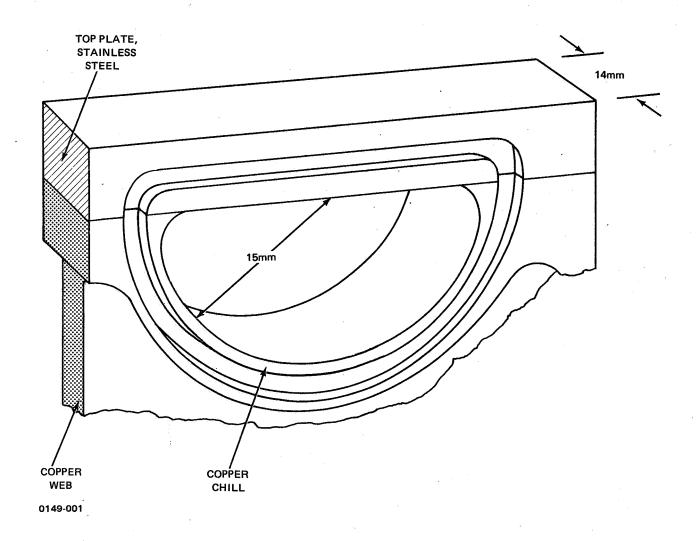
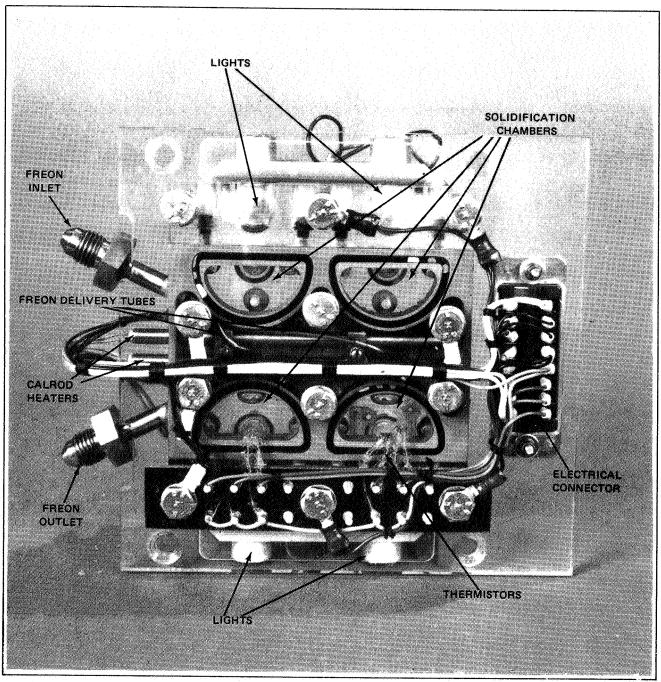


Fig. 1 Schematic Drawing of a Solidification Chamber



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Fig. 2 Overall View of the Flight-Ready Sample Holder Consisting of Four Solidification Chambers and Related Accessories

RESULTS AND DISCUSSION

LABORATORY (ONE-GRAVITY) EXPERIMENTS

Repeated laboratory simulations were performed with the flight apparatus in order to arrive at an optimum combination of the three experimentally variable parameters: solution concentrations, soak temperature, and cooling rate. The values finally chosen were such that, during one run, the solidified micro-structures varied from 25 to 100% columnar and three gravity-dependent grain multiplication mechanisms occurred. The results of these typical laboratory simulations are described in this section.

Figure 3 shows one of the lower chambers at 150s after simulated lift-off. No solid has formed yet. Since the semicylindrical wall of the sample chamber was the chill, a thermal inversion was present in this orientation. This gave rise to vigorous convection which was made visible by a simple Schlieren technique; a ruled grid had been installed on the rear face of each chamber. Also visible in Fig. 3 are three holes in the rear face of the sample chamber. The two small holes in the corners are for filling and emptying, and the larger hole communicates with the expansion chamber. The expansion chamber is a 12.5 mm diameter hole in the 12.5 mm thick plexiglass back plate. It contains a spring-loaded piston and was required to allow for expansion and contraction of the sample during heating and cooling. It provided a constant hydrostatic pressure in the sample chamber. Part of the "O" ring of the piston is visible as a black semicircular feature behind the hole.

Figure 4 shows all four chambers at 243s after simulated lift-off. White NH₄Cl crystals are present in each one. Chambers I and III contain solution A and chambers II and IV contain solution B. Chambers I and II had a completely columnar structure at this point whereas chambers III and IV had already developed a substantial equi-axed zone, in addition to the columnar growth which originated at the chill. As the experiment proceeded, columnar growth continued in II until solidification was complete whereas grain multiplication began in I and a substantial equiaxed zone formed. At the end of the experiment, columnar growth accounted for 40% of I, 100% of II, 25% of III and 50% of IV, with the remainders equiaxed. Grain multiplication occurred in III and IV via dendrite remelting caused by convection currents. The

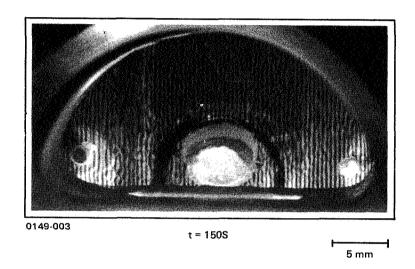
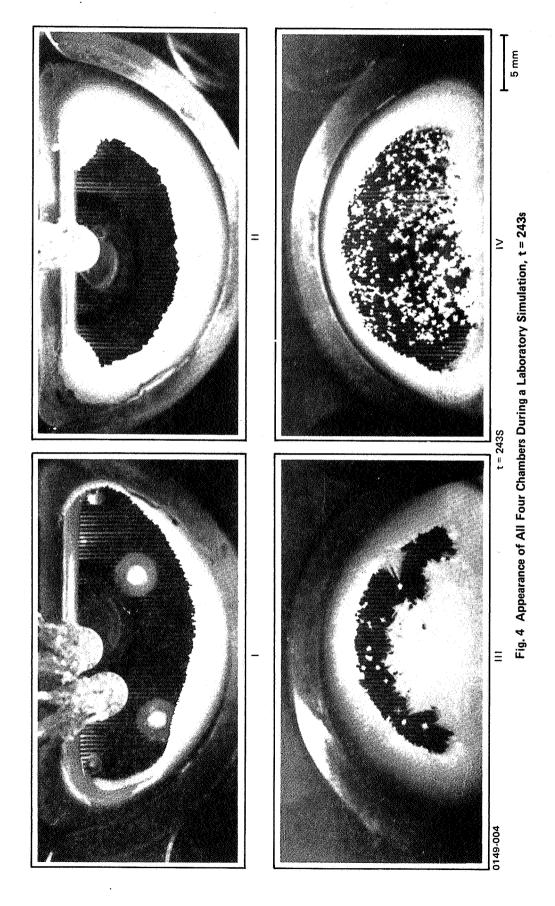


Fig. 3 Vigorous Thermal Inversion Driven Convection in IV During a Laboratory Simulation, t = 150s



convection currents were driven by thermal inversion induced density differences. The numerous crystals visible in III and IV were melted off the columnar interface by these convection currents and subsequently fell to the bottom of the chamber. In I, grain multiplication was mainly caused by showering and, to a limited extent, by convection induced dendrite arm remelting. Figure 5 shows a typical, small, grain multiplication convection cell in I. Since the chill in I was on the bottom there was no thermal inversion and thus this was a compositionally induced convection cell. As the $\mathrm{NH_4Cl:H_2O}$ solidifies in a columnar fashion, water is rejected in the interdendritic regions and the liquid therein becomes enriched in water and lighter than the liquid ahead of the dendrite tips. In the absence of complete mixing, this gives rise to a density inversion and hence, convection. The liquid which rises between the columnar dendrites accelerates dendrite arm remelting. Dendritic debris rises above the dendrite tips, leading to formation of equiaxed grains (Fig. 5). These results illustrate the effects of solute concentration and orientation of the chill (chill on top versus chill on bottom) on the extent of the columnar zone.

Figure 6 shows the results of a simulation in which the apparatus was rotated 90° so that the chill blocks were on the sides of the chambers. This orientation resulted in a highly nonuniform interface shape. Chambers II and IV were mainly columnar while I and III were mainly equiaxed. Grain multiplication occurred by convection and by showering.

LOW-GRAVITY EXPERIMENT, GENERAL OBSERVATIONS

The experiment was flown on NASA's sounding rocket flight, SPAR V. The apparatus successfully performed the experiment and the following data were obtained. Interpretation was facilitated by making the one frame per second flight film into a 16 mm cine film which could be projected at various rates. A copy of the cine film has been submitted to the program office at MSFC.

Figure 7 shows nucleation and initial growth of solid in IV.* Nine nuclei formed within seventeen seconds of each other at several locations along the chill. These crystals rapidly grew sidewise and eventually joined to form a macroscopically irregular solid-liquid interface. No evidence of Schlieren effects can be detected,

^{*}All of the photographs taken during SPAR V have a pronounced scratch which was caused by a defect in the flight camera.

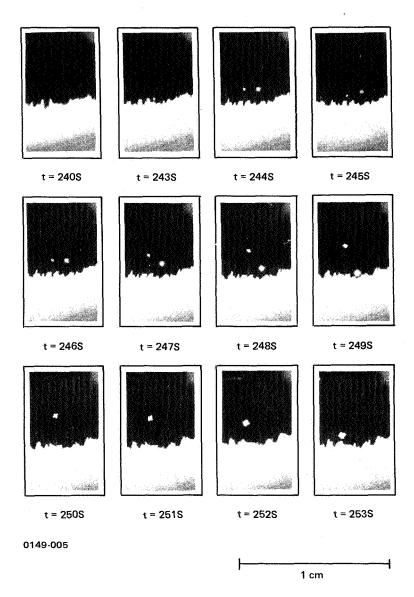


Fig. 5 Compositionally Induced Grain Multiplication Cell in Chamber I During a Laboratory Simulation

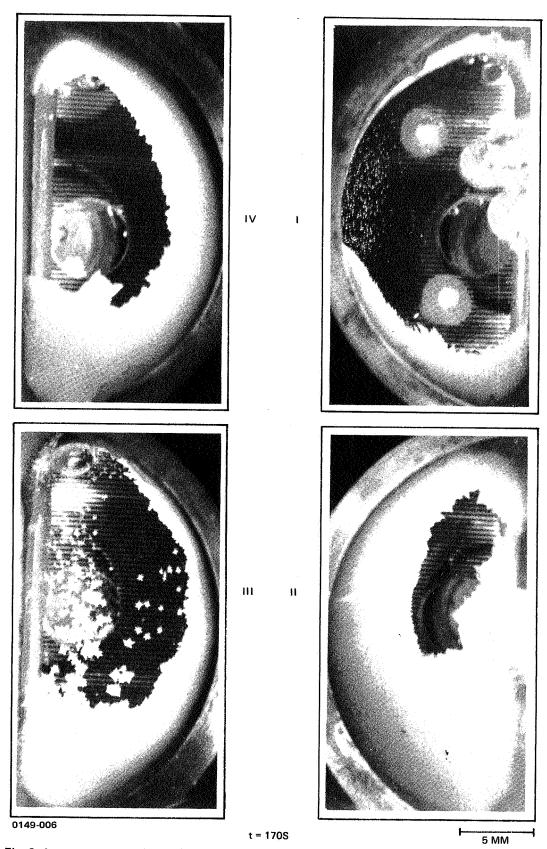
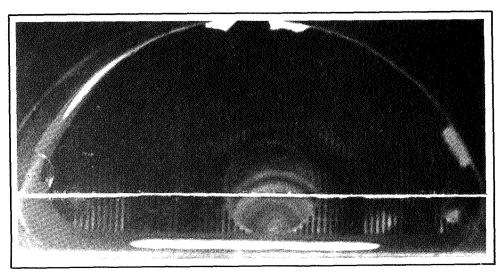


Fig. 6 Appearance of All Four Chambers During a Laboratory Simulation in Which Sample Holder was Rotated into a Vertical Orientation, t = 170s



t = 155S

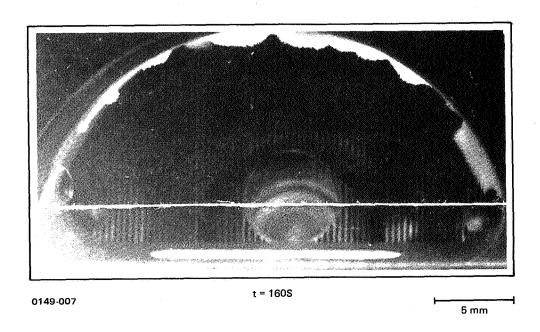


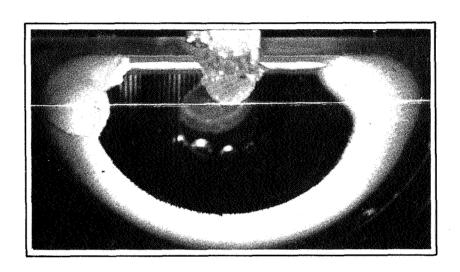
Fig. 7 Nucleation and Initial Growth in IV During Low-Gravity Experiment

thus, reduced gravity has supressed the vigorous convection observed in the laboratory simulation (Fig. 3), as expected. The irregular interface evened out as growth continued and, as seen in Fig. 8, a macroscopically smooth solid-liquid interface evolved. Comparison among Figs. 4, 6, and 8 shows the striking effect of gravitation on macroscopic interface shape. In Fig. 8 there is, of course, no difference between chill-up and chill-down positions, but, in addition, the solid-liquid interface faithfully reproduces the shape of the chill. The shape of the solid-liquid interface in the laboratory simulations (Figs. 4 and 6) was obviously influenced to a large extent by convection in the liquid. The primary dendrite spacing also appears to have been significantly more uniform in the low-gravity experiment (compare Figs. 4, 6, and 8).

Figure 9 shows the low-gravity experiment at 270s. Solidification in chambers II and IV is almost complete. All four samples solidified with a completely columnar microstructure, no columnar-to-equiaxed transition occurred, and no grain multiplication mechanisms were observed. No dendrites were observed to grow faster than the macroscopic interface, as had been reported in another low-gravity experiment (Ref. 11). Several peculiar events occurred which will be discussed in the final section. However, in order to facilitate interpretation, the thermal data will be presented now.

LOW-GRAVITY EXPERIMENT, THERMAL DATA

Thermistors had been installed in the plexiglass faces of chamber I such that their sensing elements were in intimate contact with the sample material. Two were located on the back face and two on the front, as illustrated in Fig. 4. Additional thermistors were in II and on the chill. A continuous record of the temperature as a function of time was telemetered to the tracking station during the rocket experiment. Figure 10 shows data from two of the thermistors for the flight experiment and for three different orientation ground base simulations. The chill cooled at practically the same rate in all four tests (data from the top chill test were perturbed by direct impingment of freon-12 on the chill-block thermistor), and the center of the samples cooled at almost identical rates in the three laboratory simulations. However, the center of the sample cooled at a significantly slower rate in the flight experiment. This behavior indicates that convection in the liquid accounts for a significant portion of the thermal transport in all of the laboratory simulations. This was true irrespective of the orientation of the chill. It is



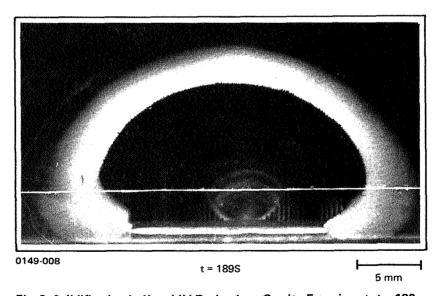


Fig. 8 Solidification in II and IV During Low-Gravity Experiment, t = 189s

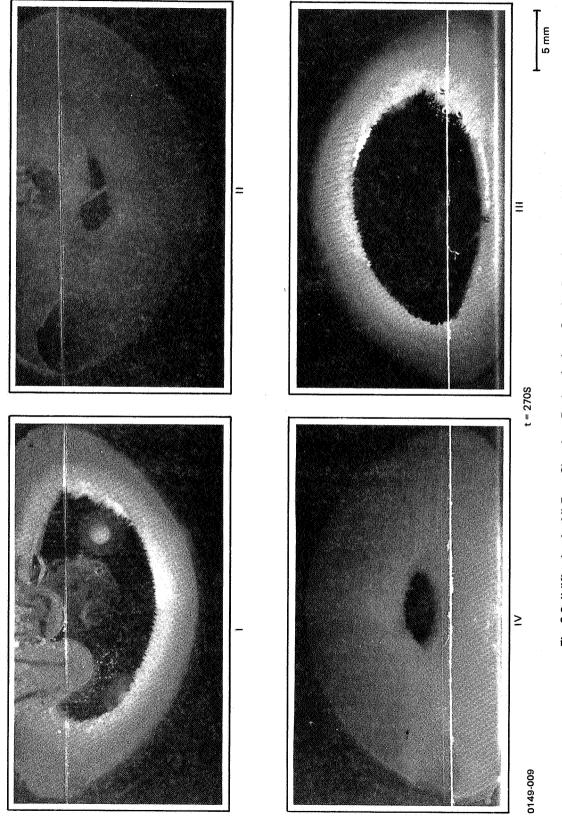


Fig. 9 Solidification in All Four Chambers During the Low-Gravity Experiment, t = 270s

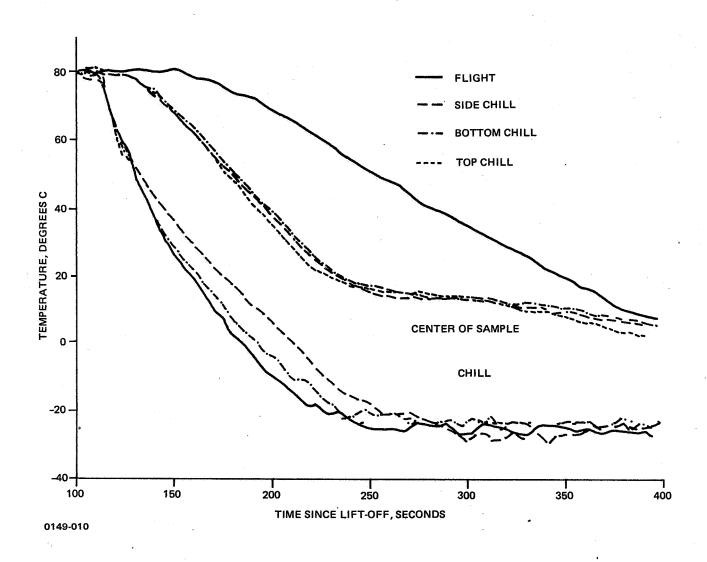


Fig. 10 Comparison of the Cooling Curves Measured at the Chill Block and Center of I During Flight and Laboratory Simulations. The Laboratory Simulations were Performed with the Sample Chamber in Three different Orientations.

thought that the semicylindrical shape of the chill was a contributory factor in this behavior; i.e., the density stratification was never purely horizontal, it was always conducive to natural convection.

The slower cooling of the center of the sample in low-gravity resulted in the presence of a steeper thermal gradient. This is illustrated in Fig. 11. It can be seen that, on a macro-scale, the thermal gradient ahead of the solid-liquid interface was significantly greater during the low-gravity experiment. It is thought that this behavior will be a general feature of low-gravity solidification operations in which the thermal gradient in the liquid is not imposed by the apparatus and heat extraction occurs through the solid.

The rate of growth of the solid was, however, not influenced by the gravity-free environment. Figure 12 shows the length of the solid measured along the center line of the front face of the sample chamber for solution A. Within experimental error all three samples grew at the same rate. The data from solution B led to a similar conclusion. Data from the side-chill experiments are not included because of the extremely nonuniform shape of the growth front. These results are to be expected since the rate of growth is controlled by thermal transport of the latent heat from the growth interface to the chill. This occurs through the solid NH₄Cl and is independent of gravitational effects.

It is the opinion of the authors that these thermal data are of particular importance in understanding the effects of gravitation on this experiment. In particular, the thermal data show that because of reduced convective transport of heat, the liquid portion of the sample was significantly warmer. This led to a steeper thermal gradient in the liquid, but more significantly, this did not allow for the growth of any equiaxed grains ahead of the columnar interface. No gravity driven convective mechanisms occurred to transport nuclei into the liquid, but this is thought to be of secondary significance. Even if nuclei had been present the thermal conditions would not have allowed them to grow. In order for an equiaxed zone to form, growth of the equiaxed grains must occur faster than growth of the columnar grains. This was not possible in the reduced gravity experiment. Support for this viewpoint can be found in several unexpected events which occurred during the flight experiment.

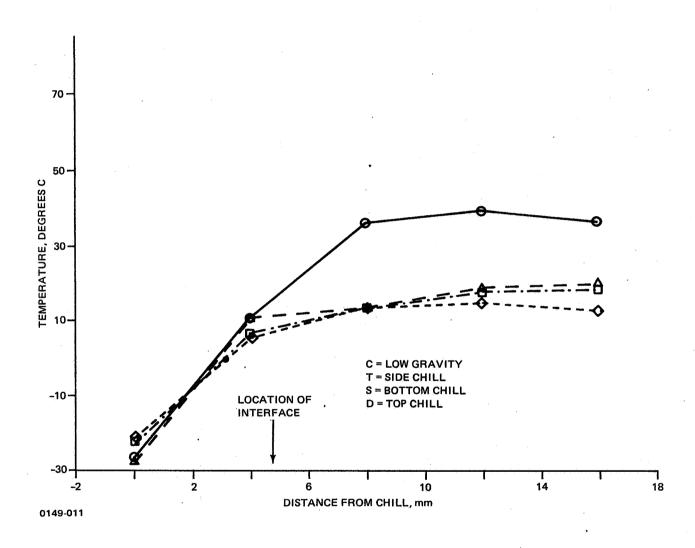


Fig. 11 The Thermal Gradient in I at t = 300s for the Flight Experiment and Three Laboratory Simulations. Location of the Solid-Liquid Interface is Marked with an Arrow.

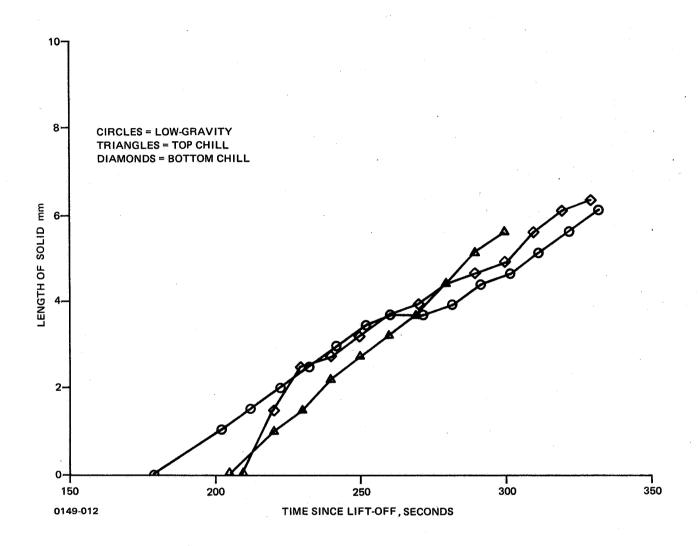


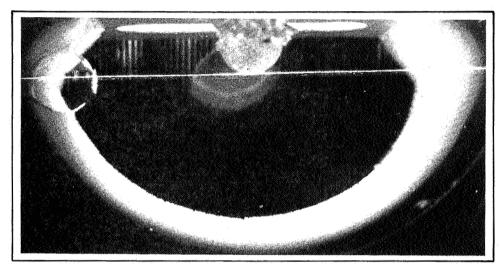
Fig. 12 Growth of NH₄Cl (Solution A) During the Flight Experiment and Two Laboratory Simulations

LOW-GRAVITY EXPERIMENT, PECULIAR EVENTS

An air bubble was present in chamber II. This bubble is presumed to have been caused by incomplete filling of the sample chamber. During solidification the bubble grew in size due to shrinkage, indicating that the expansion piston in cell II was not functioning properly. At 173s, NH₄Cl crystals appeared on the surface of the bubble. Figure 13 shows this event and subsequent growth. This seems to have been a nucleation event, but it is possible that solid simply grew along the rear surface of the bubble from existing solid. In either case, this indicates that the liquid near the surface of the bubble was cooler than liquid in the bulk. This thermal short circuit through the bubble must have been the result of an evaporation/condensation mechanism since simple gaseous conductivity should have been significantly less than the conductivity of the liquid or solid.

About 20 seconds later, another peculiar event occurred in II. Figure 14 shows this chamber at 195s and thereafter. At 195s bubbles were visible in the expansion chamber, they had been growing steadily in the 10 preceeding seconds. At 196s these bubbles disappeared and the hole in the rear face of the cell which communicates with the expansion chamber was filled with a cloud of small, opaque objects presumed to be $\mathrm{NH}_L\mathrm{Cl}$ crystals. Subsequently these crystals grew slightly and were shot out into the center of the chamber. By 200s they had arranged themselves into a saddle-shaped configuration. From then on the crystals hardly grew and the overall shape of the feature changed only slightly if at all. As solidification continued the columnar growth front simply grew past the small crystals until the feature was obliterated at about 275s. At 196s, just after the bubbles in the expansion piston collapsed, a small bubble can be seen in the center of the NH,Cl which had grown on the 4 mm bubble previously illustrated in Fig. 13. This feature is located at the top of the chamber in Fig. 14. Simultaneously, fragments of NH2Cl appeared in the liquid just ahead of the NH2Cl which had formed on the bubble surface. These observations, and repeated viewing of the cine film, led to the following reconstruction of the event.

It is presumed that the spring-loaded expansion piston was jammed, thereby allowing the pressure in the chamber to decrease and leading to bubble growth as solidification proceeded. Just after 195s the piston must have come unstuck, suddenly increasing the pressure in the chamber and causing the bubbles to collapse. Copious, shock induced, nucleation occurred in the expansion chamber and these nuclei were transported into the center of the chamber by fluid motion caused by



t = 179S

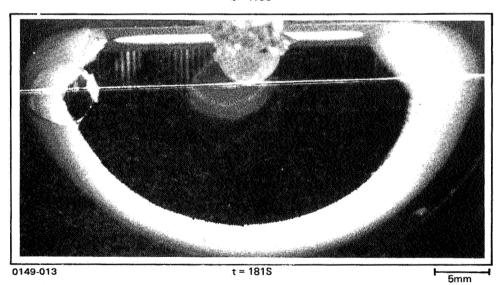


Fig. 13 Nucleation and Subsequent Growth of $\mathrm{NH_4Cl}$ on an Air Bubble in II During Flight Experiment

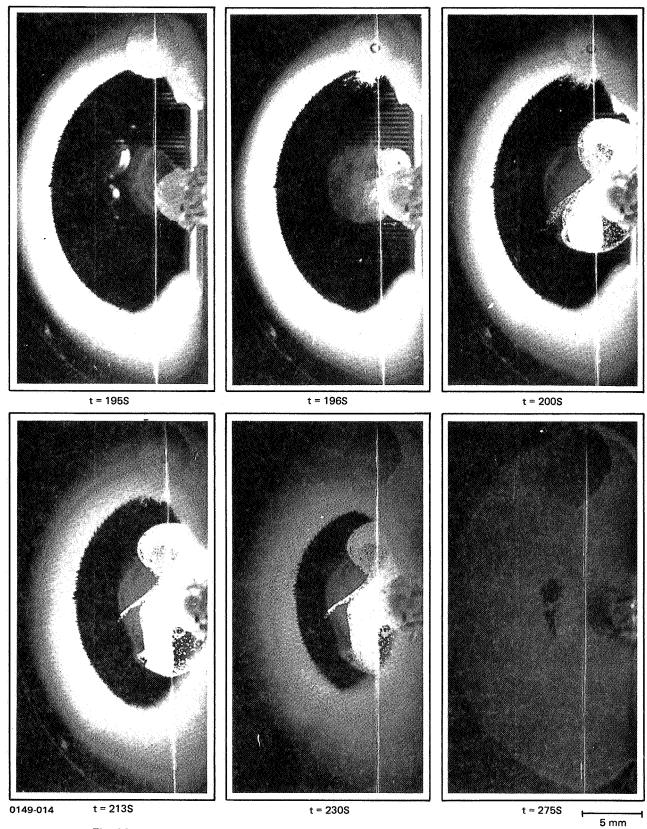


Fig. 14 "Big-Bang" Nucleation and Subsequent Growth in II During Flight Experiment

the motion of the expansion piston. The increased pressure reduced the size of the large bubble in the upper corner of the sample chamber from 4 to 0.7 mm. flected shock wave also punched out some $\mathrm{NH}_{L}\mathrm{Cl}$ dendrites from the surface of the cavity which contained this bubble and pushed them into the liquid. The sequence of events at the expansion chamber can be aptly described as "big bang" nucleation. Crystals were nucleated by a shock and transported into the liquid by fluid flow. For this experiment, however, the most important aspect of this event is the observation that although copious nuclei were formed and transported into the center of the chamber, a substantial equiaxed zone never formed. The equiaxed nuclei did not grow and did not halt the advance of the columnar interface. The columnar grains simply grew past the equiaxed ones and entrapped them. It is thought that this growth was a result of the thermal conditions existing in the low-gravity environment. This observation supports the hypothesis that, for this experiment, the primary effect of the low-gravity environment was not the supression of gravity related grain-multiplication mechanisms but, rather, the alteration of the thermal conditions by the elimination of convective thermal transport in the liquid.

Figure 15 shows a similar sequence of events which occurred in chamber I. At 214s bubbles were present in the expansion chamber. Prior frames show that they had been growing steadily. At 215s the bubbles had collapsed and a filmy, indistinct feature had appeared at the bottom of the chamber. One second later the filmy feature was more distinct; subsequent frames show that it consisted of small NH, Cl crystals which grew slightly. In addition, some small NH,Cl crystals which nucleated in the expansion chamber drifted out into the center of the solidification chamber (215-240s). They also grew only slightly, and even seem to have been shrinking at later times (275s). Their spatial arrangement did not change, again illustrating the absence of a Stokes settling force and of convection. The explanation for this sequence is thought to be similar to that postulated for chamber II; a jammed expansion piston suddenly released, causing a pressure pulse and a small amount of circulation of the melt. The pressure pulse either caused nucleation in an apparently featureless region of the melt ahead of the solidification interface or simply transported some very small dendrite fragments from the interface into the melt. Again, the most important aspect of this observation is the inability of the small equiaxed nuclei to grow, and to halt the growth of the columnar interface. The lowgravity induced modification of the thermal conditions rendered a columnar-to-equiaxed transition impossible.

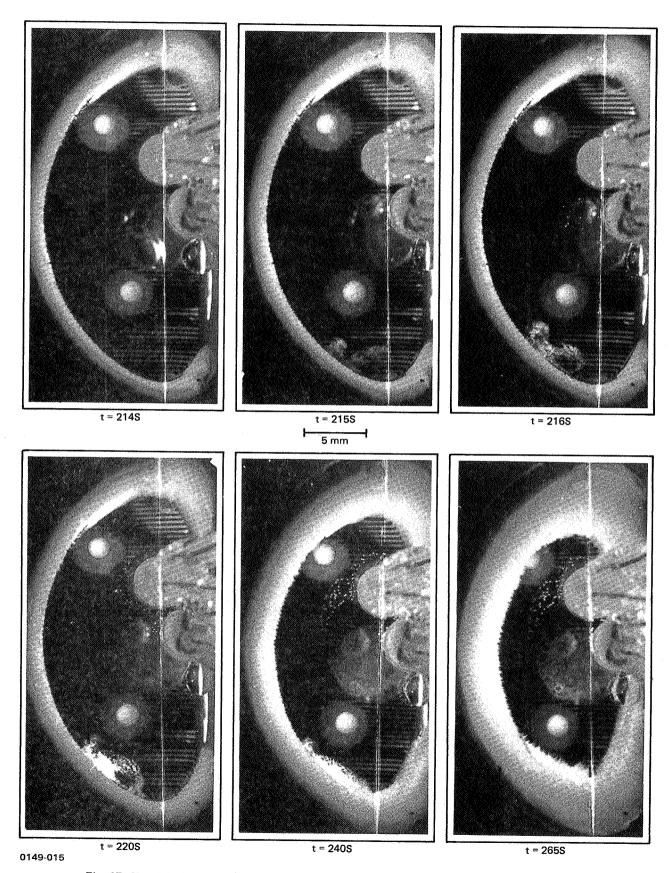


Fig. 15 Shock Induced Nucleation and Subsequent Growth in I During Flight Experiment

The last peculiar observation is shown in Fig. 16. This occurred in chamber III and involved the appearance of small crystals in an apparently featureless region of the melt (t=181), the appearance of bubbles (t=189), some small motion of bubbles, and the growth of the columnar interface past the bubbles with no apparent interaction. No complete explanation for these events is immediately obvious, but the growth of the columnar interface past these features is identical to the observations discussed previously (Figs. 14 and 15), and the lack of interaction between the dendritic interface and the bubbles is similar to previously reported low-gravity observations (Ref. 16).

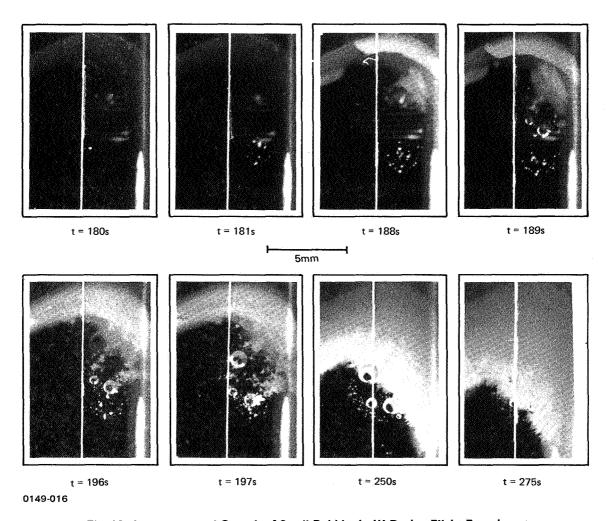


Fig. 16 Appearance and Growth of Small Bubbles in III During Flight Experiment

SUMMARY

An experimental study of the effect of a reduced gravity environment on the columnar-to-equiaxed transition in samples of 325 and 362.5 gm/litre NH_4C1 in H_2O has shown that in reduced gravity:

- Vigorous thermal inversion driven convection is absent
- No grain multiplication by showering occurs
- No thermal inversion driven grain multiplication cells occur
- No compositional inversion driven grain multiplication cells occur
- Completely columnar structures are obtained
- The liquid cools significantly slower than in one-gravity
- The thermal gradient in the liquid is steeper than in one-gravity
- The growth rate of the solid is not changed
- A bubble can act as a thermal short circuit
- Induced big bang nucleation did not give rise to an equiaxed structure
- Bubbles were not pushed by the dendritic interface

It is thought that the most significant effect of the reduced gravity environment on this experiment was the modification of the thermal conditions in the melt. The lack of convective thermal transport in the liquid resulted in conditions under which equiaxed grains could not grow and could not halt the growth of the columnar interface. The columnar-to-equiaxed transition was rendered impossible despite the presence of sufficient nuclei.

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